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For : RUBBER-REINFORCING FIBER, PROCESS FOR PRODUCING THE  
SAME, AND RUBBER PRODUCT AND PNEUMATIC TIRE EACH MADE WITH  
THE SAME

Art Unit & Examiner : 1774, Ms. Jill M. Gray

DECLARATION UNDER 37 CFR 1.132

ASSISTANT COMMISSIONER FOR PATENTS

WASHINGTON, D.C. 20231

Sir:

I, Masaaki NAKAMURA, in care of 3-2-6, Ogawahigashi-cho Kodaira-shi, Tokyo,  
Japan, declare that:

1. I graduated from Tokyo University of Science with a Bachelor's degree in School  
of Engineering in March 1991, and joined BRIDGESTONE CORPORATION in  
April 1991. Then, I have been engaged in the research and development of organic  
fiber cord-to-coating rubber adhesion in Material Development up to the present.

2. I am one of the inventors of the present U.S. Patent Application as identified  
above and familiar with the subject matter disclosed in the application.

### 3. Experiment

#### Object of Experiment

In order to clarify differences of adhesion properties between "Examples 2, 9, 10, 11, 25, 26 and 27 of this invention described in the specification" and "dry plated multi-filaments cords or short fibers made of the dry plated multi-filaments cords", the following experiments were conducted.

#### Procedure of the Experiment

The procedures for Examples 2, 9, 10, 11, 25, 26 and 27 of this invention are shown in the specification.

The multi-filaments cords of Comparative Examples A to E were made by twisting 20 of mono-filaments at 2 turns / 10cm by a twisting machine.

The same procedures for plasma cleaning and dry plating treatments as those in Example 2 were repeated to prepare Comparative Example A. The same procedures for plasma cleaning and dry plating treatments as those in Examples 9, 10, 11, 25, 26 and 27 were repeated to prepare Comparative examples B to E.

Each of the multi-filaments cords of Comparative Examples A to E was fixed in the tensile condition of 50g weight in the holder of a magnetron sputtering apparatus.

#### Test Methods

##### (1) Adhesion at 200% elongation for short fibers

The treated multi-filaments cords of Comparative Examples A to E were cut into 9 mm short fibers in length.

Each of the short fibers of Examples 2, 9, 10, 11, 25, 26 and 27 and Comparative Examples A to E was kneaded with the non-vulcanized rubber composition G-2 in a Banbury mixer and rolled into sheet, which was then vulcanized at 155 °C for 20 min under a pressure of 20 kgf/cm<sup>2</sup>. After cooling to room temperature, the vulcanized sheet was die-cut to a dumbbell shape by DIN No.

3 die so that the lengthwise direction of the dumbbell specimen extends perpendicularly to the direction to which the short fibers were made oriented by the sheeting operation.

The dumbbell specimen was subjected to a fatigue treatment by repeating 200% elongation 1000 times at 30 Hz cycle using a uniaxial fatigue tester. The specimen was re-elongated by 200% and fixed to a jig. Then, the cutting surface resulted from the dumbbell cutting was observed under a scanning electron microscope to examine the interfacial peeling between the rubber and the short fibers. Based on the results of observation, the adhesion strength between the rubber and the short fibers at 200% elongation was ranked by the following standards.

- A: No or substantially no peeling
- B: Slight peeling
- C: Peeling or break due to peeling

(2) Adhesion at 200% elongation for twisted cords

Each of the treated multi-filaments cords of Comparative Examples A to E was inserted between the sheets of the non-vulcanized rubber composition G-2 and the each rubber sheet having the multi-filaments cords both side edges of which were secured at constant length, was then vulcanized at 155 °C for 20 min under a pressure of 20 kgf/cm<sup>2</sup>. After cooling to room temperature, the vulcanized sheet was die-cut to a dumbbell shape by DIN No. 3 die so that the lengthwise direction of the dumbbell specimen extends perpendicularly to the direction of the multi-filaments cords. The multi-filaments cords of Comparative Examples A to E were twisted at about 0.1 turns / the width of the dumbbell specimen so that they were without the filaments convergence.

The tests were conducted in the same procedure as (1) above mentioned.

(3) Bead filler rubber - short-fiber adhesion

The short fibers of Example 2 were kneaded with the non-vulcanized rubber composition G-2 in Banbury mixer, and rolled into sheet to prepare a short

fiber-compounded rubber. A pneumatic tire was prepared and tested as described in the specification.

**(4) Bead filler rubber - twisted cord adhesion**

The treated multi-filaments cords of Comparative Examples A were cut by 3 cm in length. Then, the multi-filaments cords were put in the non-vulcanized rubber composition G-2. The preparation of pneumatic tires and the tests were conducted in the same procedure as (3) above mentioned.

**Result**

The results obtained are shown in the following Table 1.

Table 1-1

	Example 2	Comparative Example A	Example 9	Comparative Example B	Example 10	Comparative Example C
Fiber Material raw material kind single fiber diameter ( $\mu$ m)	Polyester F-3 103	Polyester F-3 twisted multi-filaments cord	Polyarylate F-5 45	Polyarylate F-5 twisted multi-filaments cord	Nylon F-6 125	Nylon F-6 twisted multi-filaments cord
Rubber Composition	G-2	G-2	G-2	G-2	G-2	G-2
Evaluation Results (1) Adhesion at 200% elongation for short fibers	A	C	A	C	A	C
(2) Adhesion at 200% elongation for twisted cords		C		C		C
(3) Bead filler rubber - short fiber adhesion	A					
(4) Bead filler rubber - twisted cord adhesion		C				

Table 1-2

	Example 11	Comparative Example D	Example 25	Example 26	Example 27	Comparative Example E
Fiber Material	Aramid	Aramid	glass	glass	glass	glass
raw material kind	F-7	F-7	I-4	I-5	I-6	I-4
single fiber diameter ( $\mu$ m)	100	twisted multi-filaments cord	9	9	10-24	twisted multi-filaments cord
Rubber Composition	G-2	G-2	G-2	G-2	G-2	G-2
Evaluation Results						
(1) Adhesion at 200% elongation for short fibers	A	C	A	A	A	C
(2) Adhesion at 200% elongation for twisted cords		C				C

4. Consideration

It is clearly recognized from the results of the above Experiments that the adhesion properties of the short fibers of Examples 2, 9, 10, 11, 25, 26 and 27 of this invention are far superior to those of the multi-filaments cords and the short fibers made of the multi-filaments cords of Comparative Examples A to E.

5. I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

July 13 2006

Date

Masaaki Nakamura

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